Integrated Computational Materials Engineering (ICME)

Predictive Tools Development for Low Cost Carbon Fiber for Lightweight Vehicles

- 2018 Annual Merit Review -

PI / Presenter: Xiaodong (Chris) Li, University of Virginia

June 19, 2018

Project ID: MAT124

Overview

Timeline

- Start Date: October 1, 2017
- End Date: September 30, 2020
- Percent Complete: 18%

Budget

- *Total* Project Funding: $4,408,032
  - $3,000,000 Federal
  - $418,032 Cost Share
  - $990,000 LightMat Consortium
- *FY 2018* Funding: $1,452,478
  - $985,134 Federal
  - $137,344 Cost Share
  - $330,000 LightMat Consortium

Barriers

- Reduction of vehicle weight necessitates lower-density materials with suitable mechanical properties, low-cost carbon fiber
- Development of a calibrated ICME predictive tool that can identify & optimize fiber processing parameters
- Extend the ICME Framework to encompass synthesis and characterization of fibers based on alternative precursors and novel manufacturing processes

Partners

- University of Virginia *(Lead)*
- Pennsylvania State University
- Oak Ridge National Laboratory
- Solvay S.A.
- Oshkosh Corporation

University of Virginia Engineering

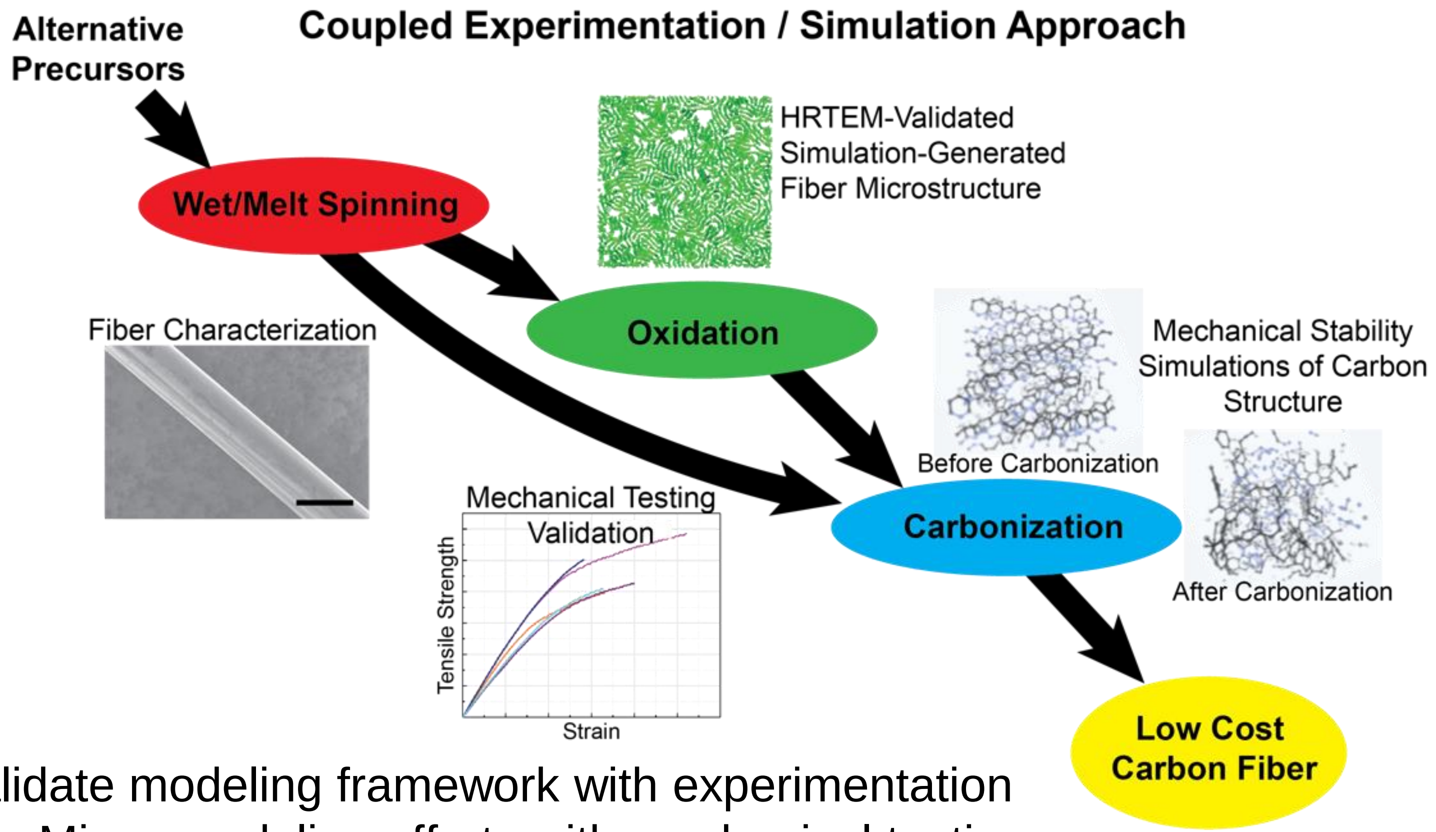
Relevance

- **Primary objective** to demonstrate CF precursor technology and processing techniques capable of achieving the following:
    - Cost ≤ $5/pound
    - Strength ≥ 250 Ksi
    - Modulus ≥ 25 Msi
    - Strain ≥ 1%
- This objective will be accomplished through the ICME framework, coupling simulations and targeted experimentation to evaluate alternative precursors for suitability to manufacture low-cost CF
- First fiscal year (FY) objectives:
    - Statistical analysis of PAN precursor oxidation and carbonization
    - Chemical conversion of PAN-based carbon fibers (simulation + experimental verification)
    - Fiber and fiber/matrix mechanics
    - Wet/melt spinning optimization
- **Impact** - Accelerate the capability of significant vehicle weight reduction and high-strength material systems, while developing a technical framework to enable at-scale production.

Any proposed future work is subject to change based on funding levels.

Innovative Approach & Strategy

- Assemble a framework to model conversion of fibers and predict properties
  - Model individual processing steps, including oxidation and carbonization, to predict coupled thermal-chemical-mechanical fiber transformation



- Validate modeling framework with experimentation
  - Mirror modeling efforts with mechanical testing and chemical characterization of fibers before and after each processing step

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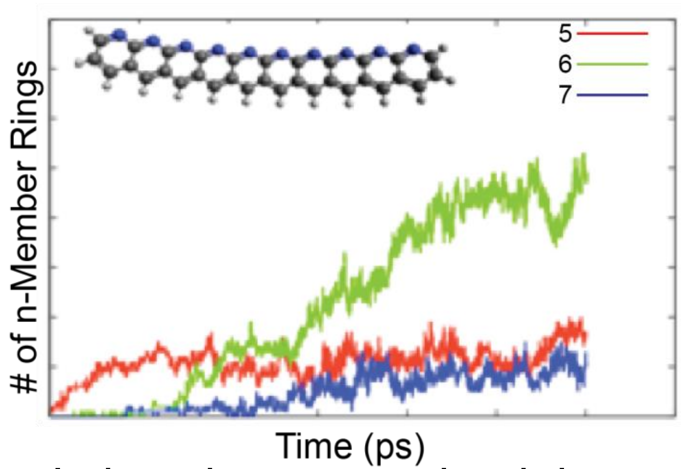
Approach and FY18 Milestones

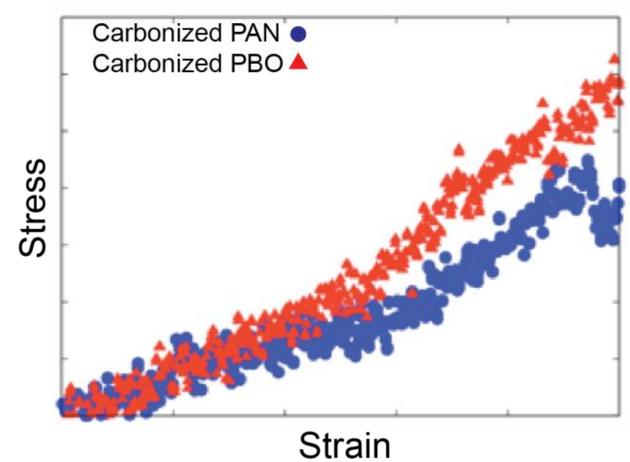
- The project is on track to meet FY18 milestones

Date	Milestone	Status
September 30, 2018	**M1:** Statistical analysis of PAN precursor oxidation, baseline reference for comparison with statistical results of thermomechanical testing laboratory synthesized fibers.	**On-Track 80%**
September 30, 2018	**M2:** Chemical conversion of PAN-based carbon fibers and verification through direct comparison with M1. Milestone will be met if simulations correctly identify resultant properties within 15% error margin.	**On-Track 60%**
September 30, 2018	**M3:** Fiber and fiber/matrix mechanics correctly predicted by ICME simulations as compared to resultant properties and characteristics of synthesized fibers.	**On-Track 60%**
September 30, 2018	**M4:** Wet/melt spinning optimization for preliminary production-scale synthesis able to reproduce laboratory-scale fiber properties at-scale.	**On-Track 80%**
September 30, 2018	**Go/No Go M5:** Accurate prediction of PAN-fiber properties. Simulations will estimate PAN-based fiber properties (strength, modulus, strain) within a 15% error margin. Results will be verified with mechanical testing of the fibers to confirm a go-decision.	**On-Track**

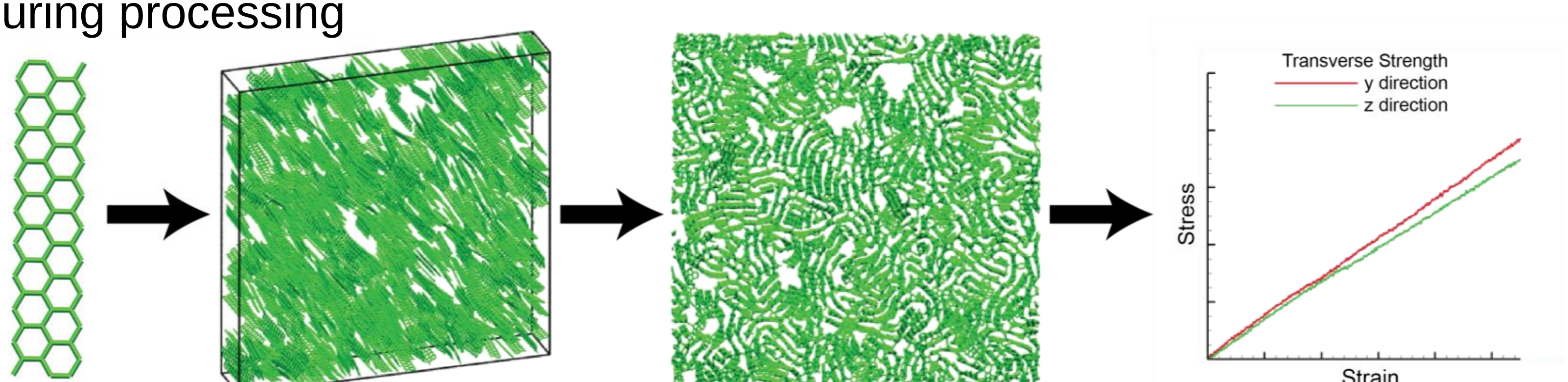
Technical Accomplishments

- ReaxFF simulations have been developed to quantify bond energies resulting from chemical transformations during carbonization and to estimate fiber strength from the structure of the carbonized fiber





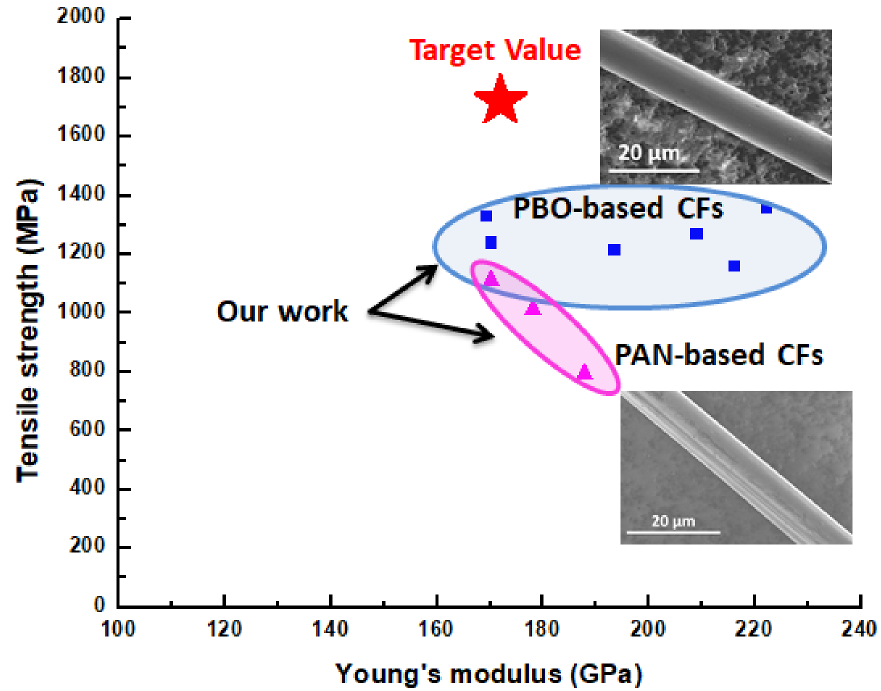
- MD simulations have examined the transformation of the fiber microstructure during processing



Carbon Ladder | Ladder units within representative periodic volume | MD-NPT Produced Stable Fiber Structure | Simulation Property Predictions based on Microstructure

Technical Accomplishments

- Lab-scale fiber synthesis used to validate simulation results and identify alternative manufacturing procedures
- Exploration of alternative precursors and production methods, including introduction of nanostructured precursors and reduced oxidation temperatures
- Optimization of manufacturing parameters which influence CF properties



Potential Stages for Fiber Production Optimization

Alternative Precursors	Wet/Melt Spinning	Oxidation	Carbonization	Low Cost Carbon Fiber
- Less expensive materials - Informed selection based on desired microstructure and chemistry - Enhancements with dispersed nanostructures	- Smart control of spinning parameters, including strain rate, temperature	- Optimize oxidation temperatures and duration - Cut out oxidation entirely - Smart control of fiber tension	- Optimize carbonization temperatures and duration - Smart control of fiber tension	- Optimized mechanical performance - Minimal energy consumption

Any proposed future work is subject to change based on funding levels.

Responses to Previous Year Comments

- This project began October 2017; it was not reviewed previously

Collaboration and Team Coordination

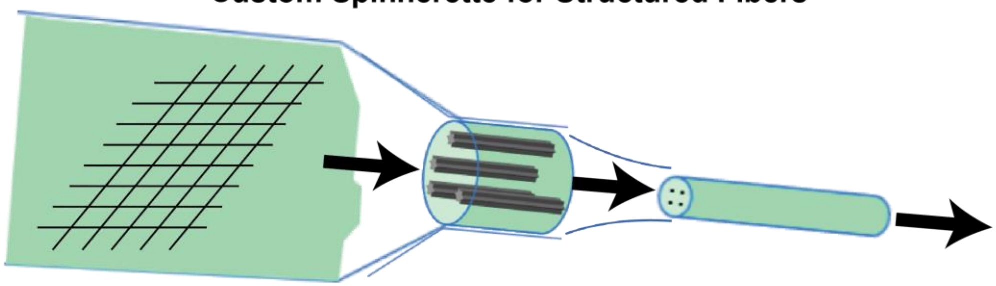
- **UVa** Co-PI Zhigilei & **Penn State** Co-PI van Duin — Subcontractors
  - Developing simulations of the chemical conversion of precursor fibers and large-scale MD simulations of fiber chemical-mechanical behavior
- **Oak Ridge National Laboratory** — Subcontractor
  - Experimental analysis of alternative precursors and large-scale pilot runs
- **Solvay S.A.** — Subcontractor
  - Industry guidance on fiber characterization techniques and development of laboratory-scale PAN production
- **Oshkosh Corporation** — Subcontractor
  - Industry insight on unique constraints and priorities for technology transfer from research laboratory to industrial production

Remaining Challenges and Barriers

Key Challenges

- **Challenge:** Limitation of lab-scale production techniques
  - Lab-scale fiber spinning procedures need further optimization to maximize properties of PAN fibers
  - Custom equipment (spinnerette) found necessary for uniform fiber diameters and are being installed

Custom Spinnerette for Structured Fibers



Structure within Spinnerette to create nano-filamets within fiber

Precursor with ligamentous structure leaving nozzle

Filament with fibrils during drawdown

- **Challenge:** Compatibility of PAN-ICME framework to alternative precursors
  - Key parameters will require recalibration to new polymers to link characterized fibers to prediction framework

Any proposed future work is subject to change based on funding levels.

Proposed Future Research

Future Work

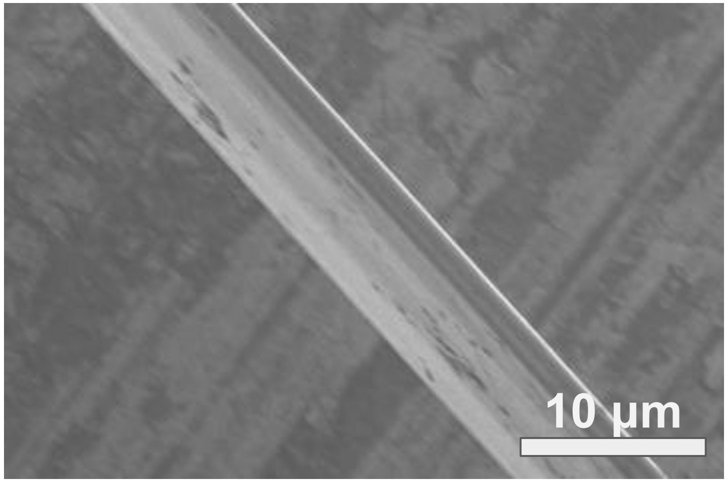
- **FY18 -** Successful, accurate prediction of PAN-fiber properties with ICME framework
  - Optimization of computational framework
  - Framework validation with experimental testing of PAN fibers
- **FY19 -** Framework adapted to predict alternative fiber precursors
  - Statistical analysis of alternative precursor stabilization
  - Understanding the chemical conversion of new precursors
  - Synthesis and characterization of prototype precursor
- **FY20 -** Computational framework integrated into testbed with 15% accuracy of production scaling strategies
  - Scalability/economics study of carbon fiber production

Any proposed future work is subject to change based on funding levels.

Summary

- ReaxFF simulations have been developed to quantify bond energies, resulting from chemical transformations during carbonization, in order to predict fiber structure and estimate fiber strength
- MD simulations have examined the transformation of the PAN fiber microstructure during oxidation and carbonization processing
- Lab-scale PAN fiber synthesis has been used to validate simulation results and identify alternative manufacturing procedures
- Alternative PBO fiber has been explored to decrease manufacturing energy consumption by eliminating the oxidation process and by reducing the carbonization temperature
- These achievements put us on track to **succeed**!

PAN Fiber



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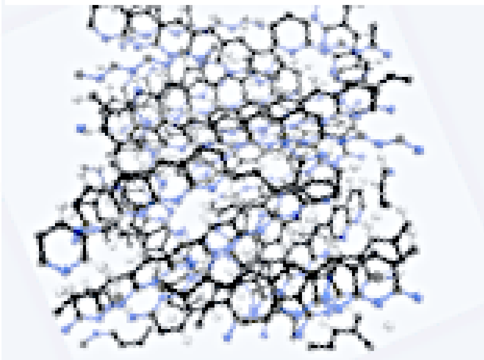
Technical Backup Slides

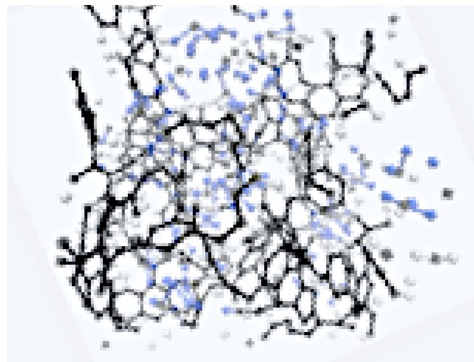
Technical Backup Slides – PAN Fibers

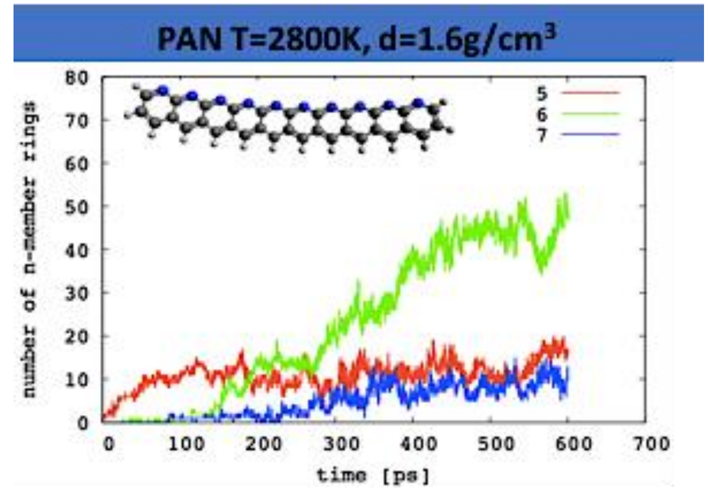
- Per first FY objectives, PAN is used as a preliminary material to establish the ICME framework
- ReaxFF simulations on PAN carbon structure transformation during carbonization

Before Carbonization PAN

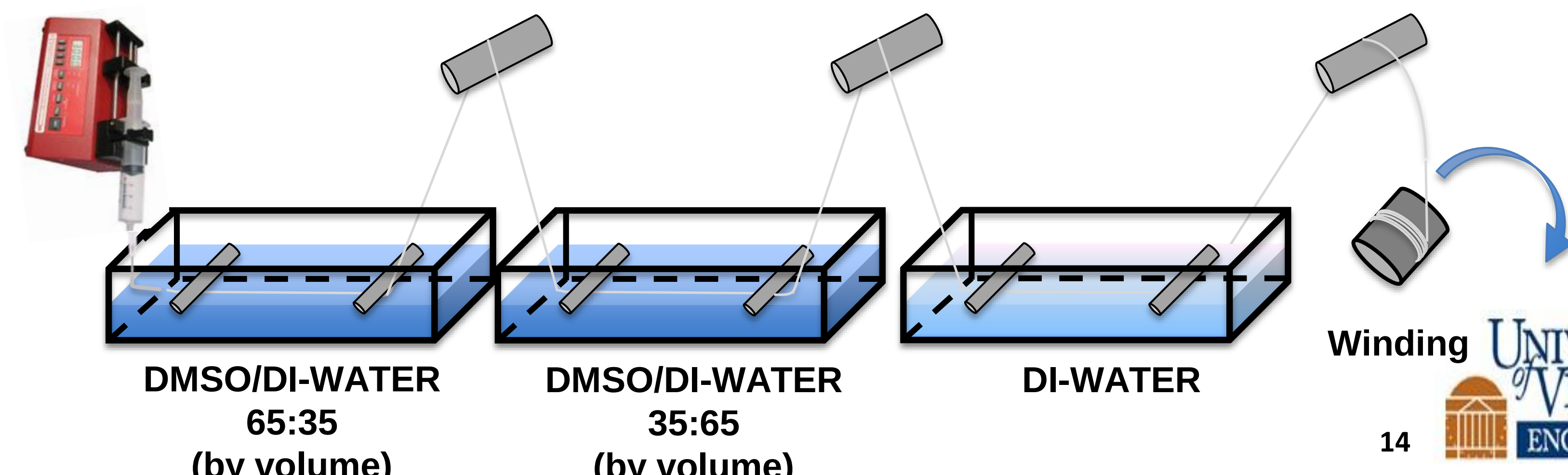


Carbonized PAN





- Simulations validated with experimental production of PAN fiber



Technical Backup Slides – MD Structural Models

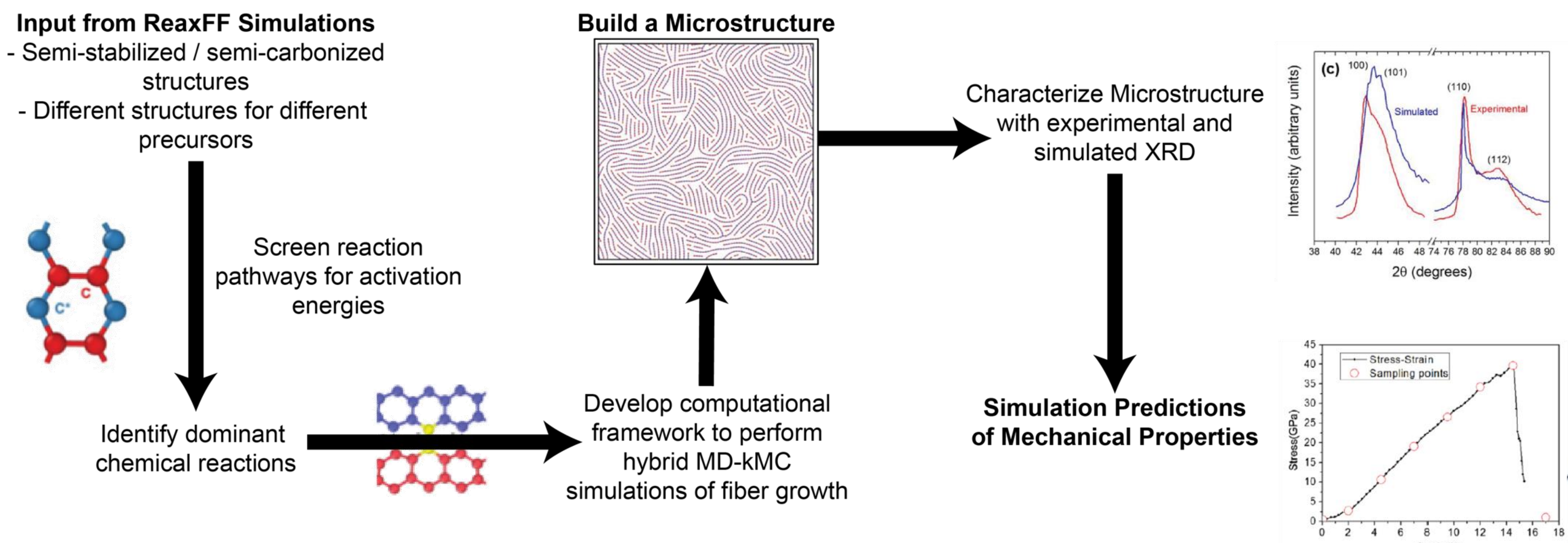
- Large-scale atomistic modeling of structure and mechanical properties of carbon fibers

Precursor → CF Microstructure

- Identify dominant reaction chemistry
- Design a novel MD-kMC method
- MD-kMC simulations to obtain microstructure

Microstructure → Mechanical Properties

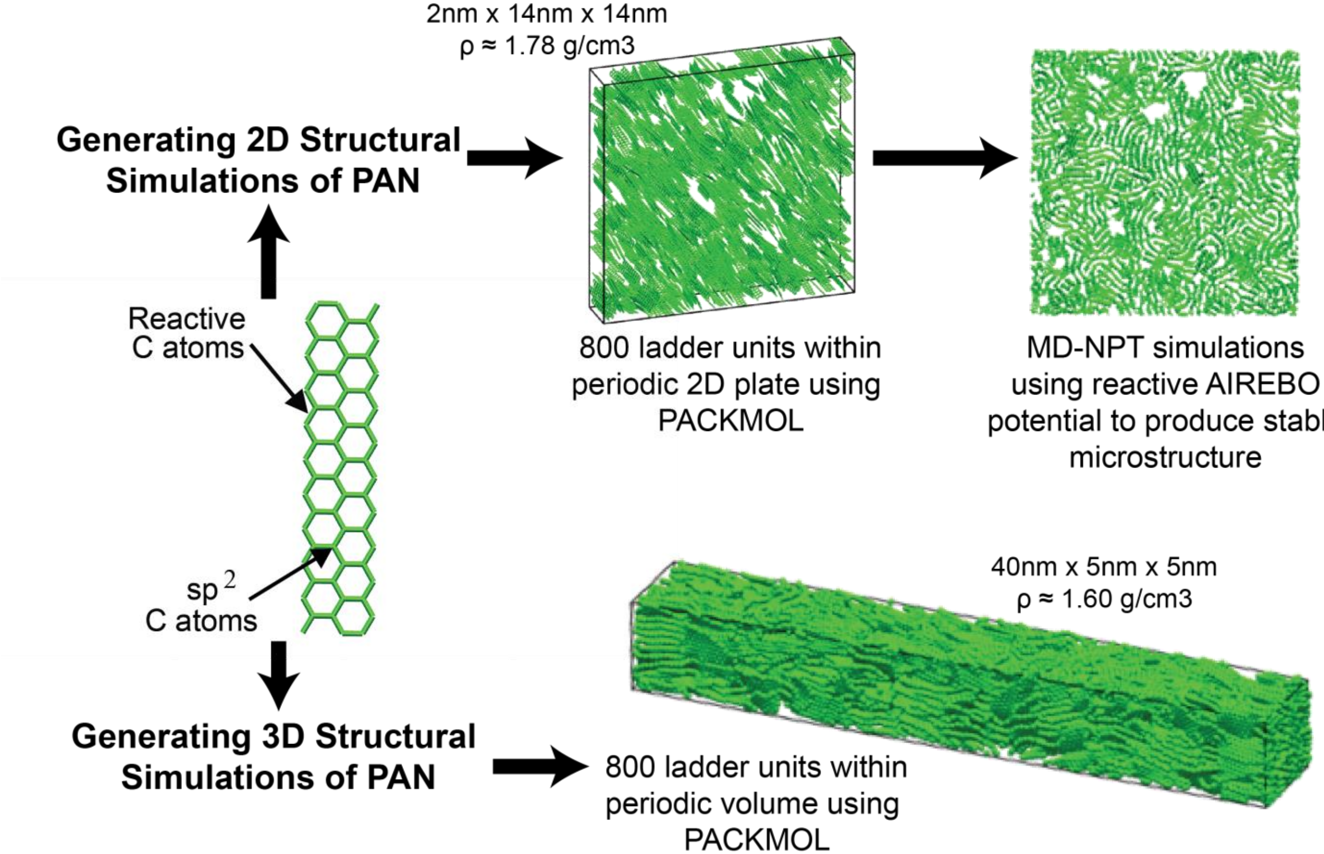
- Microstructure characterization
- Large-scale MD simulations of mechanical deformation
- Identify key structural features that control mechanical properties and draw connection back to the precursor



1. Desai, Li, Shen, Strachan; J. Chem. Phys.; 147; 224705; 2017
2. Gupta, Artyukhov, Penev, Yakobson; Adv. Mater.; 28; 10317; 2016

Technical Backup Slides – MD Structural Models

- Simulations have been developed to build 2D and 3D fiber microstructures for simulated mechanical testing



- These simulations accurately predict fiber modulus
  MD-Predicted: 12.7-14 GPa **Experimental**: 9-15 GPa, nanoindentation[1]

1. Maurin, R., Davies, P., Baral, N., Baley, C.; Appl. Comp. Mat.; 2008; 15; 61

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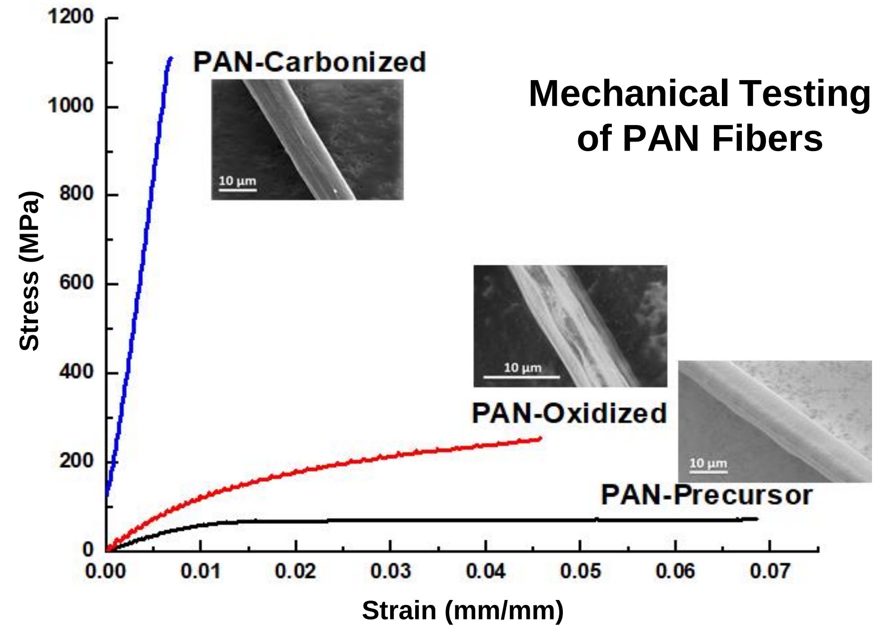
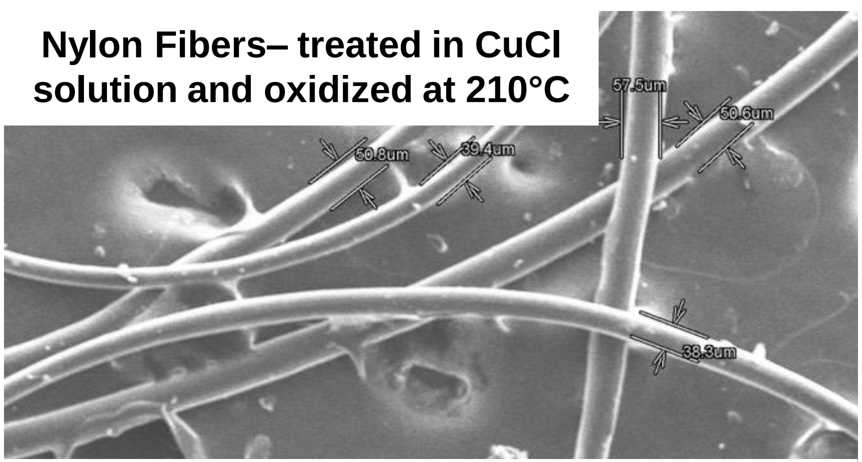
Technical Backup Slides - Experimentation

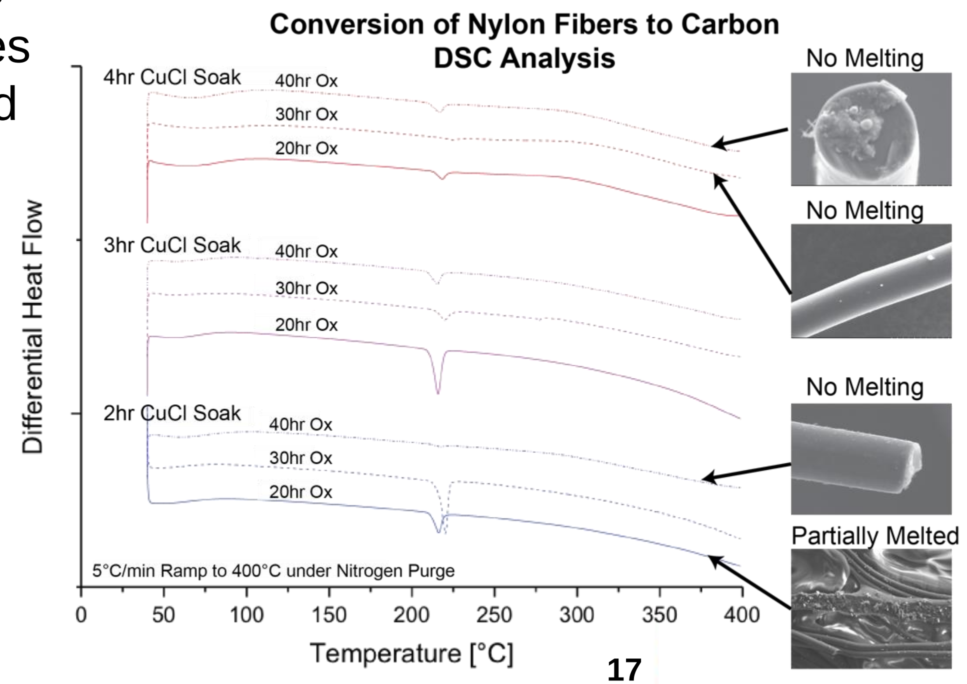
- Experimental mechanical testing is used to validate model predictions of CF properties
- Experimentation of CF production parameters and alternative precursors will also pre-screen materials
- Preliminary experimentation with low cost nylon fibers shows great promise and fibers with dispersed nanoparticles (*ex*. graphene) demonstrate enhanced modulus



Nylon Fibers– treated in CuCl solution and oxidized at 210°C



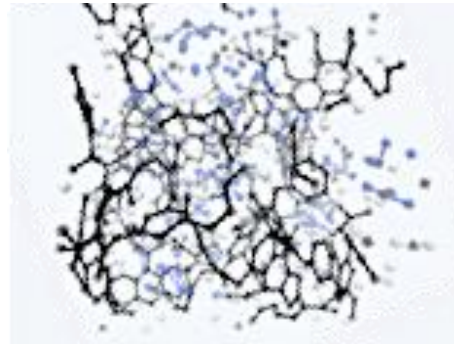
Nylon fiber work supported by LightMat Consortium



Technical Backup Slides - PBO Fibers

- PBO is also a potential precursor
- ReaxFF simulations of PBO carbonization identify few additional carbon rings but more pronounced clustering, which leads to superior mechanical stability compared to PAN
- Experimental results revealed PBO fibers may be produced without oxidation and at a substantially lower carbonization temperature, signaling a production technique for carbon fiber with significant energy/cost savings

Carbonized PAN



Carbonized PBO